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Synthesis, structure and antimicrobial activity of dialkyl [(hydroxy)(4-nitrophenyl)methyl]phosphonates

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Table of Contents

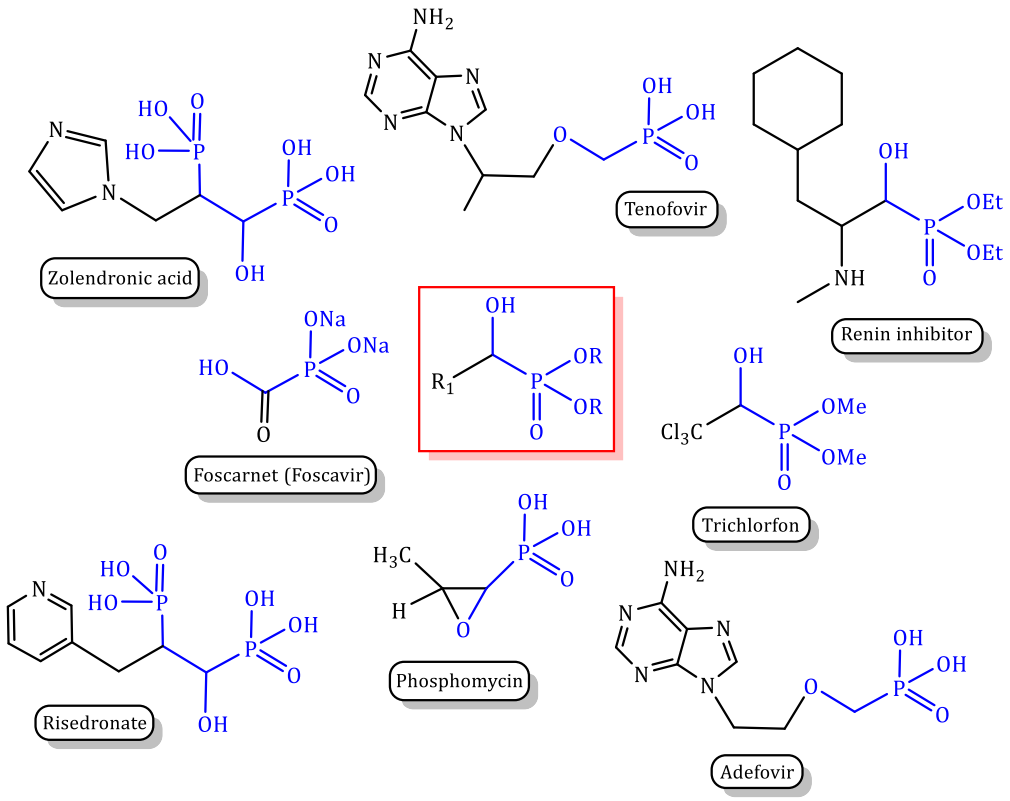


Figure S1 Examples of bioactive α-hydroxy phosphonates

I. Experimental

All reagents used in the synthesis were of the highest quality available. Commercially available solvents were purified by standard procedures. Dialkyl phosphites were obtained according by the standard methods. Potassium phosphate 97.5 % were used.

NMR spectra were recorded at room temperature in $CDCl_3$ solution on a Bruker Avance III 400 spectrometer with an operating frequency of 162 MHz for ^{31}P spectra, an operating frequency of 400 MHz for ^{1}H spectra and an operating frequency of 100.6 MHz for ^{13}C spectra in a solution of $CDCl_3$.

The Fourier Transformed InfraRed (FTIR) spectrum of the sample was recorded using Perkin Elmer UATR Two FT-IR Spectrometer (Spectrum Two, USA) with an ATR (attenuated total reflectance) diamond crystal. Spectra were baseline corrected and normalized.

Simultaneous thermogravimetry and differential scanning calorimetry (TG/DSC) analysis of solid samples were performed using the thermoanalyzer STA 449F1 Jupiter (Netzsch, Germany) at the temperature range of 40-300 °C. In each experiment, the temperature rate was 10 °C/min, and an argon atmosphere with a total flow rate of 75 ml/min was used.

The X-ray diffraction data for the single crystals **1**, **2** and **4** were collected on a Bruker AXS D8 Venture diffractometer at temperature T=120(2) K. Data collection was performed according to recommended strategies employing an ω/φ-scan mode. The programs used: APEX3 for data collection, SAINT for data reduction, SADABS for multi-scan absorption correction, SHELXT for structure solution, SHELXL for structure refinement by full-matrix least-squares against F2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms at carbon atoms were placed into calculated positions and refined as riding atoms. CCDC 2375061-2375063 contain the supplementary crystallographic data for this paper.

The antimicrobial activity was determined in vitro using the agar disk-diffusion method using Mueller-Hilton agar medium against a variety of pathogenic microorganisms in EtOH: *Staphylococcus aureus* (ATCC 29213), *Bacillus cereus* (ATCC 11778), as well as with Gram-negative cultures: *Escherichia coli* (ATCC 25922), *Pseudomonas aeruginosa* (ATCC 27853), *Candida albicans* (ATCC 10231)

General procedure for the synthesis of dialkyl [(hydroxy)(4-nitrophenyl)methyl]phosphonates 1-4

The synthesis was carried out in a round-bottomed flask equipped with a magnetic stirrer. *p*-Nitrobenzaldehyde (0.02 mol) was poured into the flask, then 0.02 mol of the corresponding dialkyl phosphites was added. Then 0.002 mol of potassium phosphate (catalyst) was added. The reaction was carried out at room temperature without solvent. The progress of the reaction was monitored using FTIR spectroscopy and ^{31}P NMR. After completion of the reaction, the reaction mixture was dissolved in 5 ml of dichloromethane and filtered. The solvent was distilled off on a rotary evaporator, the dry residue was recrystallized from hexane and filtered on a Schott funnel, then washed with hexane.

Diethyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate 1.

Yield 94%, yellow powder. mp 90.2 ± 0.1 ˚C. FTIR spectrum, cm^{-1}: 1024 (P-O-C), 1237 (P=O), 1347, 1518 (NO_2), 3238 (OH). ^{1}H NMR ($CDCl_3$), δ_H, ppm.: 1.20 t. (3H, C<u>H</u>$_3$, $^3J_{HH}$ 7.08 Hz), 1.22 t. (3H, *C<u>H</u>$_3$, $^3J_{HH}$ 7.08 Hz), 3.98 – 4.11 m. (4H, OC<u>H</u>$_2$), 5.14 d. (1H, PC<u>H</u>, $^2J_{PH}$ 13.09 Hz), 5.93 s. (1H, O<u>H</u>), 7.62 d. (2H, Ar-H, $^3J_{HH}$ 8.95 Hz), 8.14 d. (2H, Ar-H $^3J_{HH}$ 8.59 Hz). ^{13}C{^{1}H} NMR ($CDCl_3$), δ_C, ppm.: 16.17 d. (<u>C</u>H$_3$, $^3J_{PC}$ 4.84 Hz), 16.22 d. (*<u>C</u>H$_3$, $^3J_{PC}$ 4.97 Hz), 63.14 d. (<u>C</u>H$_2$, $^2J_{PC}$ 7.81 Hz), 63.81 d. (*CH$_2$, $^2J_{PC}$ 6.87 Hz), 69.77 d. (P<u>C</u>H, $^1J_{PC}$ 160.05 Hz), 123.05 d. ($^3J_{PC}$ 2.53 Hz), 127.62 d. ($^2J_{PC}$ 5.20 Hz), 144.59 s., 147.26 s. (Ar). ^{31}P{^{1}H} NMR ($CDCl_3$): δ_P 19.8 ppm.

The spectral data of compound **1** also have been described [S1].

Diisopropyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate 2.

Yield 92%, yellow powder. mp 105.3 ± 0.1 ˚C. FTIR spectrum, cm^{-1}: 991 (P-O-C), 1233 (P=O), 1341, 1517 (NO_2), 3194 (OH). ^{1}H NMR ($CDCl_3$), δ_H, ppm.: 1.16 – 1.26 m. (12H, C<u>H</u>$_3$), 4.61 – 4.76 m. (2H, OC<u>H</u>), 5.11 d. (1H, PC<u>H</u>, $^2J_{PH}$ 12.55 Hz), 5.50 s. (1H, O<u>H</u>), 7.66 d. (2H, Ar-H, $^3J_{HH}$ 8.90 Hz), 8.19 d. (2H, Ar-H, $^3J_{HH}$ 8.87 Hz). ^{13}C{^{1}H} NMR ($CDCl_3$), δ_C, ppm.: 23.64 d. (<u>C</u>H$_3$, $^3J_{PC}$ 5.18 Hz), 23.83 d. (*<u>C</u>H$_3$, $^3J_{PC}$ 3.77 Hz), 23.94 d. (**<u>C</u>H$_3$, $^3J_{PC}$ 4.74 Hz), 24.04 d. (***<u>C</u>H$_3$, $^3J_{PC}$ 3.07 Hz), 70.23 d. (P<u>C</u>H, $^1J_{PC}$ 159.80 Hz), 72.12 d. (O<u>C</u>H, $^2J_{PC}$ 7.90 Hz), 72.66 d. (O<u>C*</u>H, $^2J_{PC}$ 7.60 Hz), 123.06 d. ($^3J_{PC}$ 2.70 Hz), 127.75 d. ($^2J_{PC}$ 5.10 Hz), 144.72 s., 147.33 s. (Ar). ^{31}P{^{1}H} NMR ($CDCl_3$): δ_P 18.1 ppm.

Dibutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate 3.

Yield 93%, yellow powder. mp 71.5 ± 0.1 ˚C. FTIR spectrum, cm^{-1}: 1026 (P-O-C), 1238 (P=O), 1349, 1516 (NO_2), 3415 br (OH). ^{1}H NMR ($CDCl_3$), δ_H, ppm.: 0.75 – 0.95 m. (6H, C<u>H</u>$_3$), 1.20 – 1.40 m. (4H, C<u>H</u>$_2$CH$_3$), 1.45 – 1.65 m. (4H, OCH$_2$C<u>H</u>$_2$), 3.93 – 4.10 m. (4H, OC<u>H</u>$_2$), 5.17 d. (1H, PC<u>H</u>, $^2J_{PH}$ 12.60 Hz), 5.71 s. (1H, O<u>H</u>), 7.65 d. (2H, Ar-H, $^3J_{HH}$ 8.50 Hz), 8.19 d. (2H, Ar-H, $^3J_{HH}$ 8.30). ^{13}C{^{1}H} NMR ($CDCl_3$), δ_C, ppm.: 13.42 s. (<u>C</u>H$_3$), 18.51 d. (OCH$_2$<u>C</u>H$_2$, $^3J_{PC}$

6.20 Hz), 32.36 s. ($CH_3\underline{C}H_2$), 66.76 d. ($O\underline{C}H_2$, $^2J_{PC}$ 7.60 Hz), 67.58 d. ($O\underline{C^*}H_2$, $^2J_{PC}$ 7.30 Hz), 69.95 d. ($P\underline{C}H$, $^1J_{PC}$ 159.40 Hz), 123.13 s., 127.64 d. ($^2J_{PC}$ 5.20 Hz), 144.65 s., 147.36 s. (Ar). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ_P 19.7 ppm.

Diisobutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate 4.

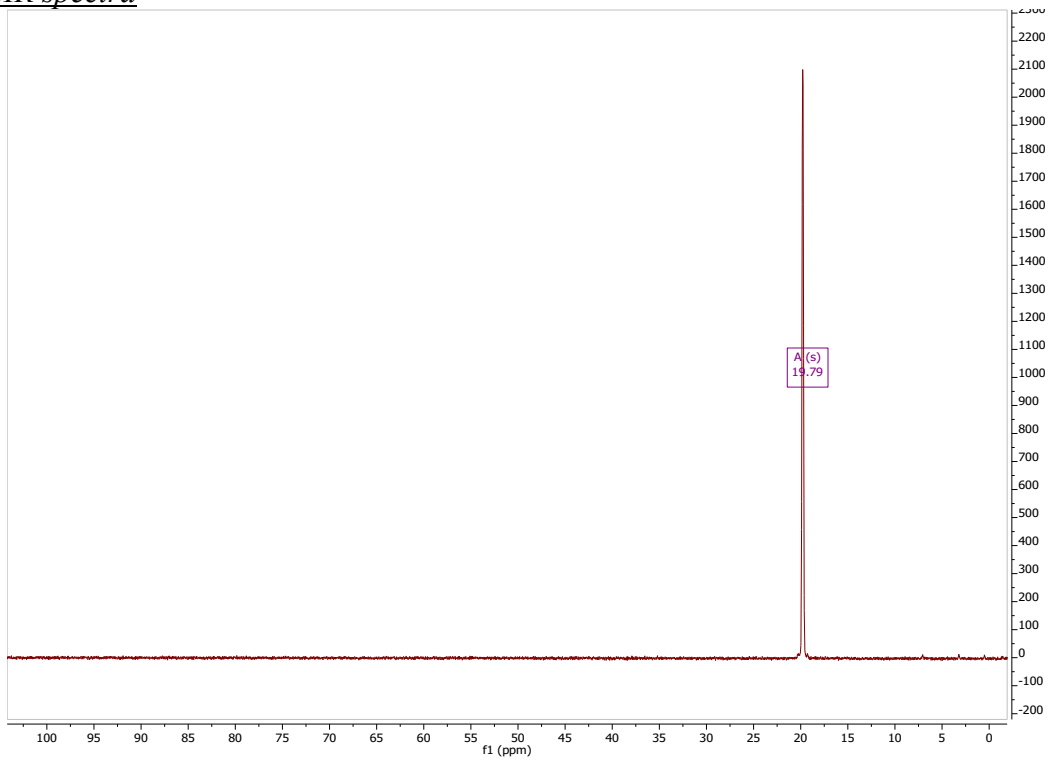
Yield 95%, yellow powder. mp 113.3 ± 0.1 ˚C. FTIR spectrum, cm^{-1}: 1011 (P-O-C), 1221 (P=O), 1346, 1518 (NO_2), 3225 (OH). ^1H NMR ($CDCl_3$), δ_H, ppm.: 0.77 – 0.90 m. (12H, $C\underline{H}_3$), 1.76 – 1.92 m. (2H, $OCH_2C\underline{H}$), 3.72 – 3.86 m. (4H, $OC\underline{H}_2$), 5.19 d. (1H, $PC\underline{H}$, $^2J_{PH}$ 12.60 Hz), 5.96 s. (1H, $O\underline{H}$), 7.65 d. (2H, Ar-H, $^2J_{HH}$ 8.80 Hz), 8.17 d. (2H, Ar-H, $^2J_{HH}$ 8.80 Hz). $^{13}C\{^1H\}$ NMR ($CDCl_3$), δ_C, ppm.: 18.46 s. ($\underline{C}H_3$), 29.13 s. ($OCH_2\underline{C}H$), 69.98 d. ($P\underline{C}H$, $^1J_{PC}$ 159.60 Hz), 72.77 d. ($O\underline{C}H_2$, $^2J_{PC}$ 8.00 Hz), 73.64 d. ($O\underline{C^*}H$, $^2J_{PC}$ 7.70 Hz), 123.08 d. ($^3J_{PC}$ 2.80 Hz), 127.69 d. ($^2J_{PC}$ 5.20 Hz), 144.70 s., 147.33 s. (Ar). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ_P 19.3 ppm.

References

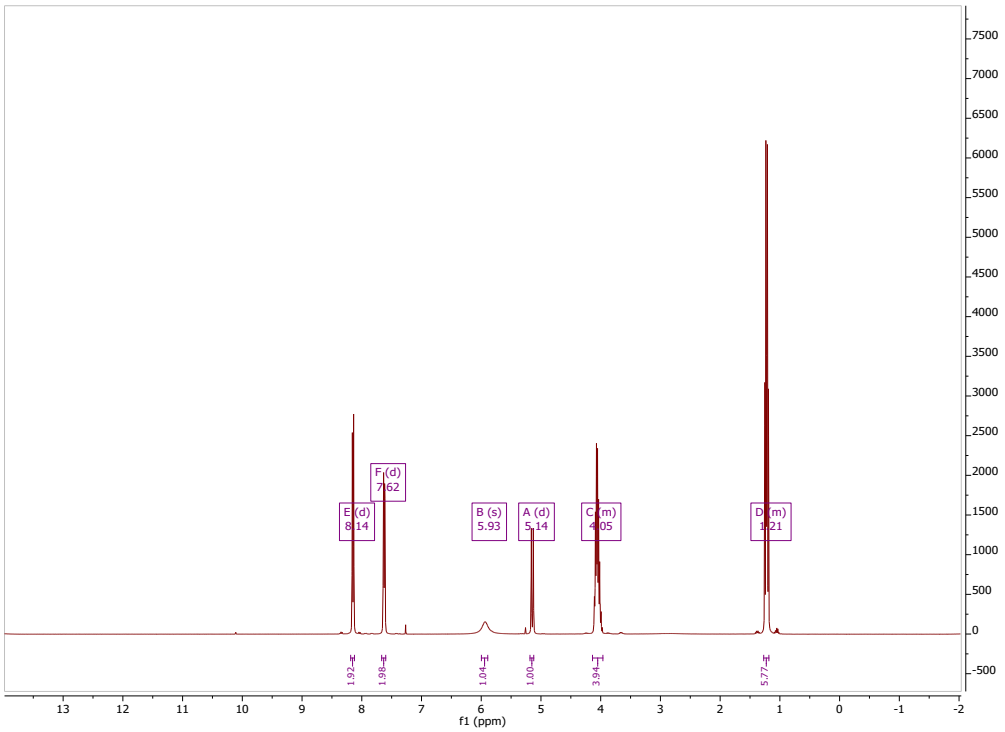
S1. G.Keglevich, R. T. Viola and D. Laszo, *Heteroatom Chemistry*, 2011, **22**, 15; https://onlinelibrary.wiley.com/doi/abs/10.1002/hc.20649.

II. Spectral data and TG/DSC curves for dialkyl [(hydroxy)(4-nitrophenyl)methyl]phosphonates 1-4

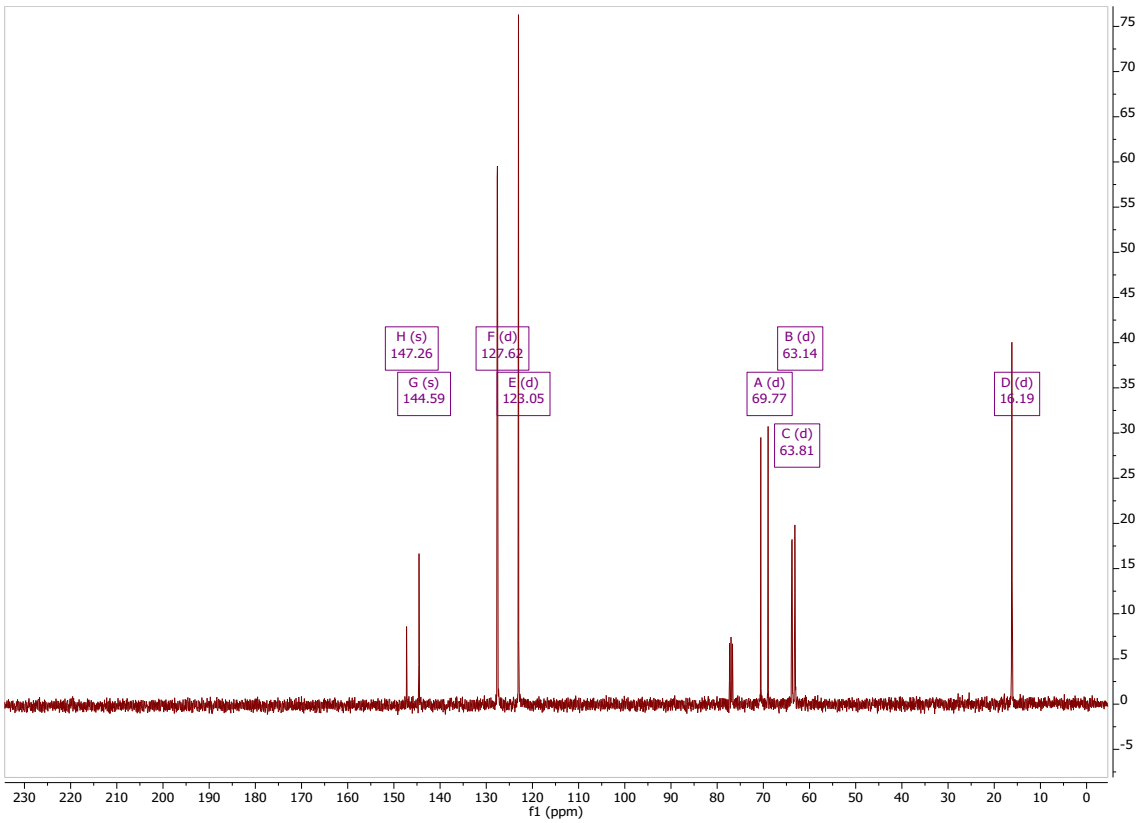
NMR spectra



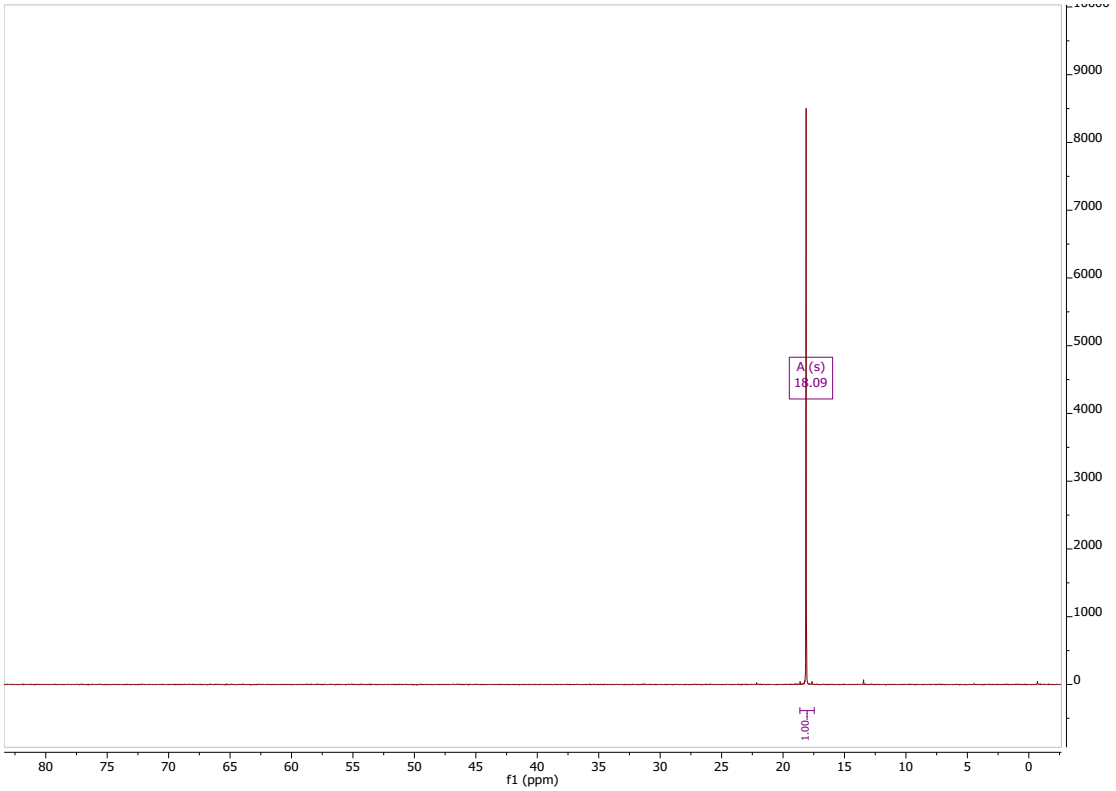
$^{31}P\{^{1}H\}$ NMR ($CDCl_3$) of diethyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **1**



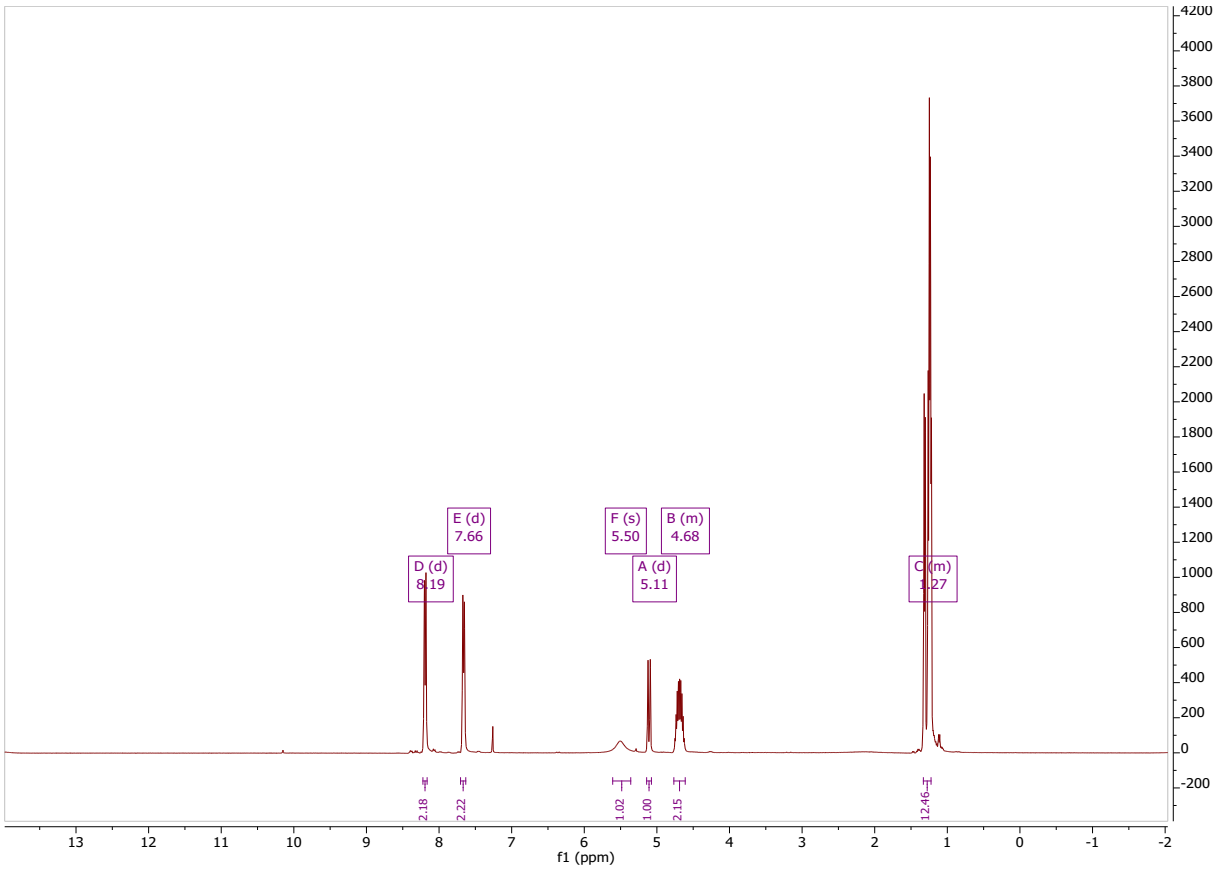
^{1}H NMR ($CDCl_3$) of diethyl[(hydroxy)(4-nitrophenyl)methyl]phosphonate **1**



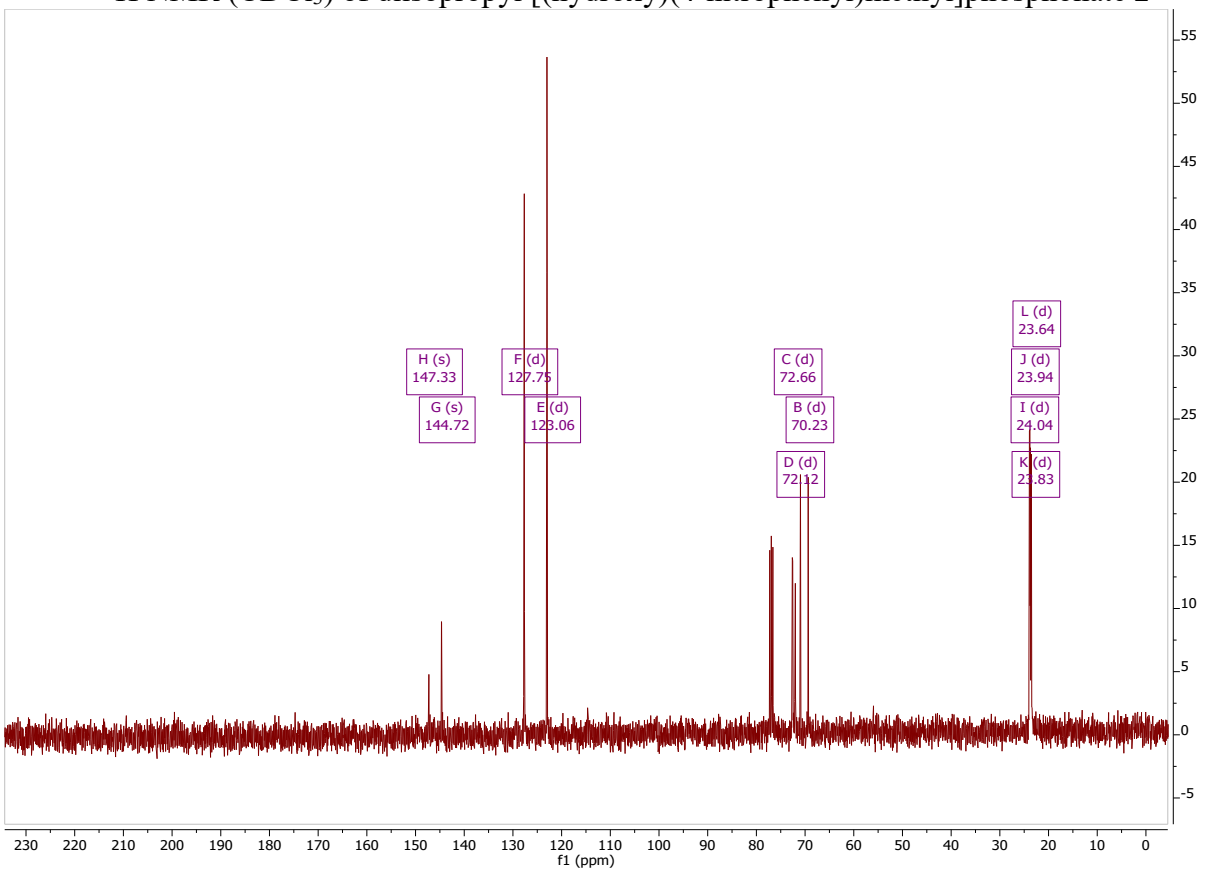
$^{13}C\{^{1}H\}$ NMR ($CDCl_3$) of diethyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **1**



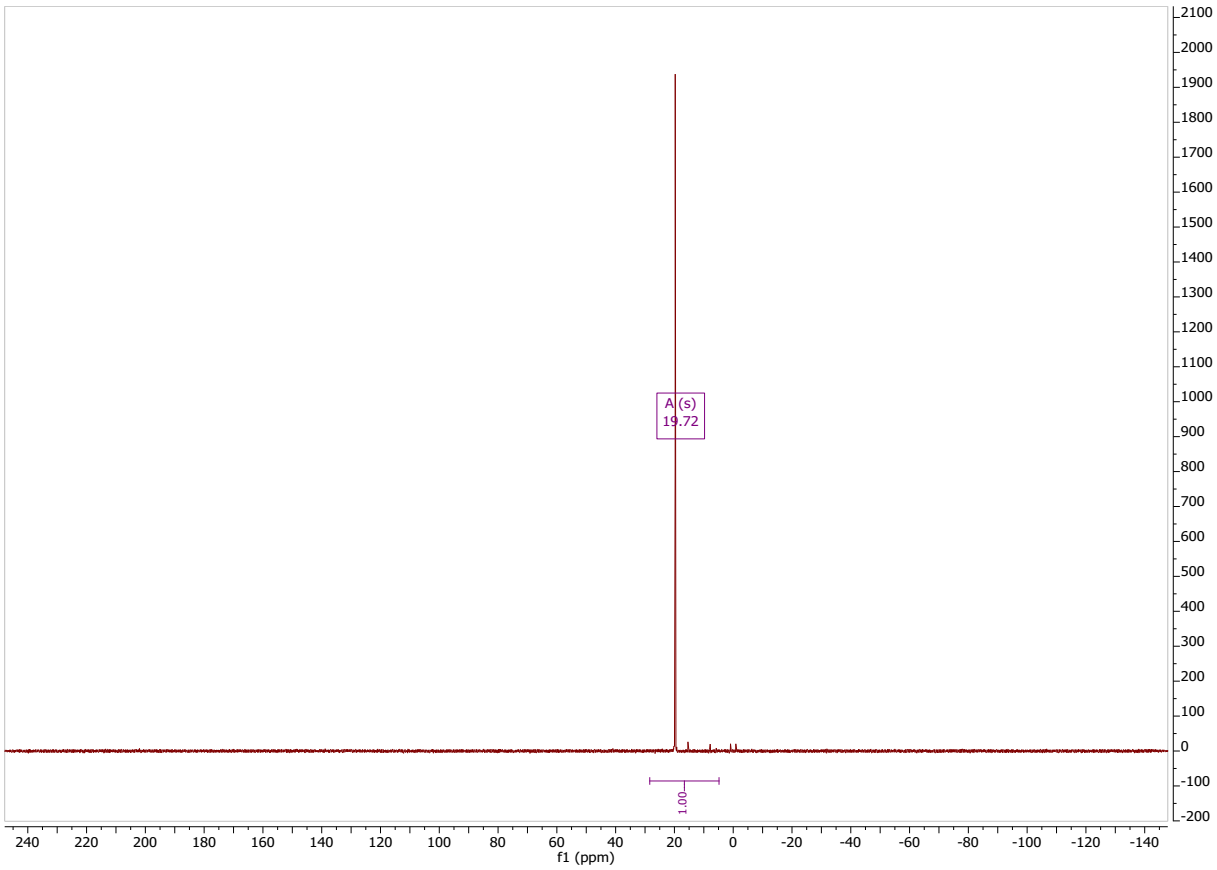
$^{31}P\{^{1}H\}$ NMR ($CDCl_3$) of diisopropyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **2**



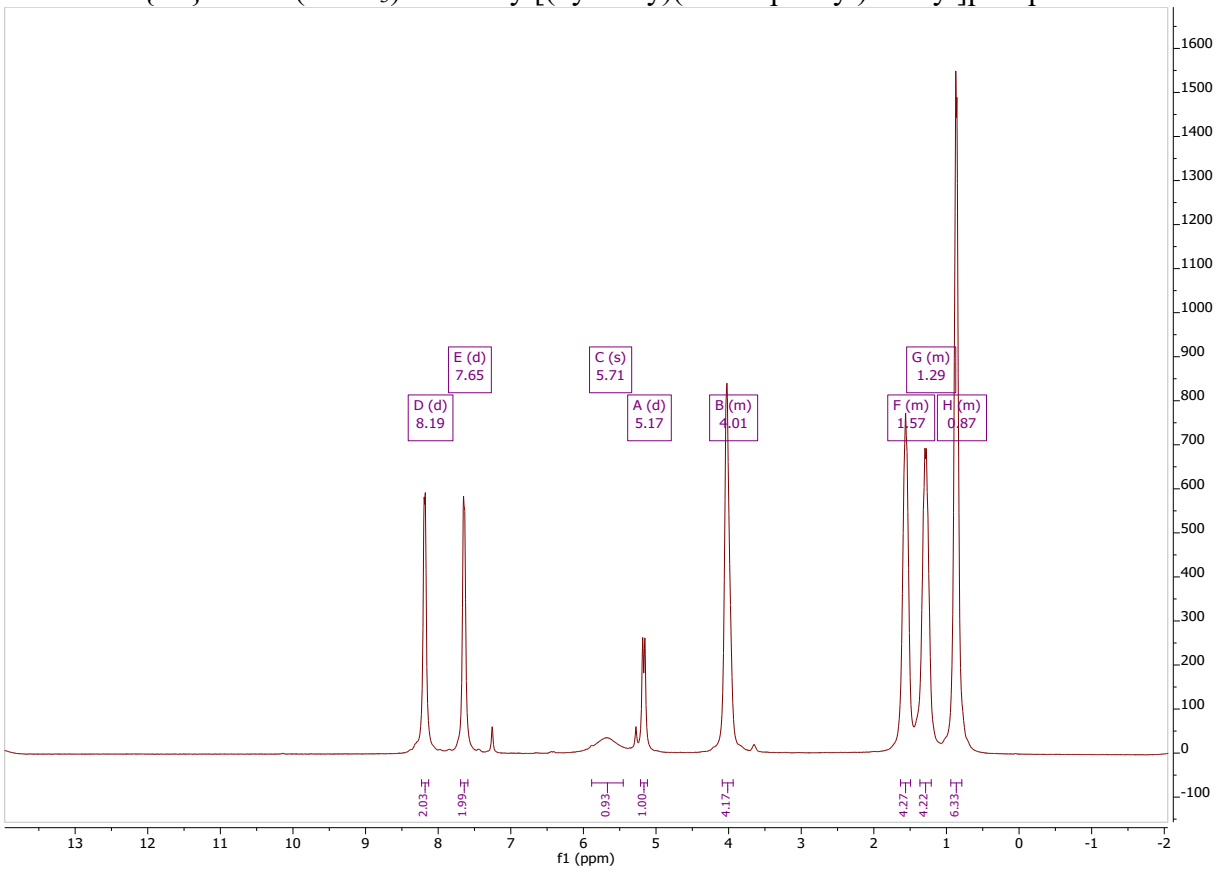
^{1}H NMR ($CDCl_3$) of diisopropyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **2**



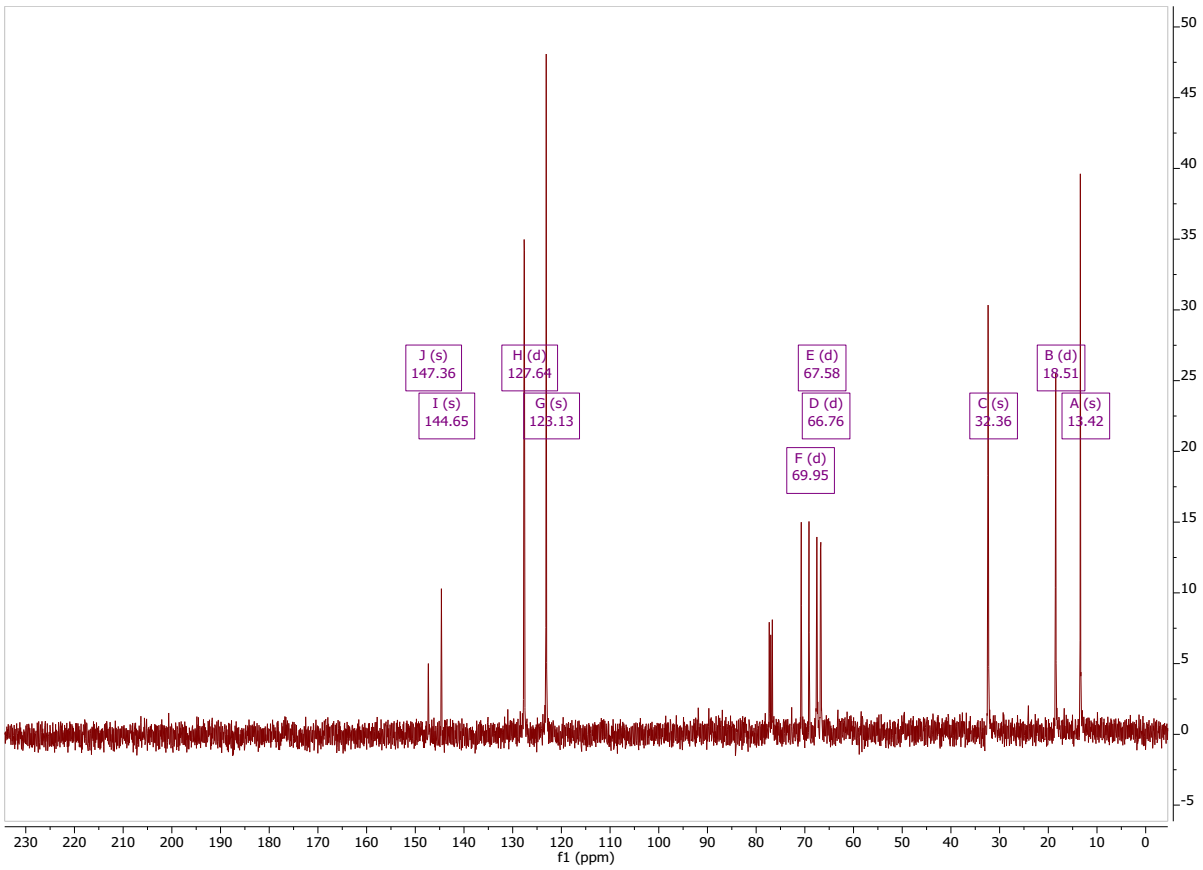
^{13}C{^{1}H} NMR ($CDCl_3$) of diisopropyl [(hydroxy)(4-nitrophenyl)(hydroxy)methyl]phosphonate **2**



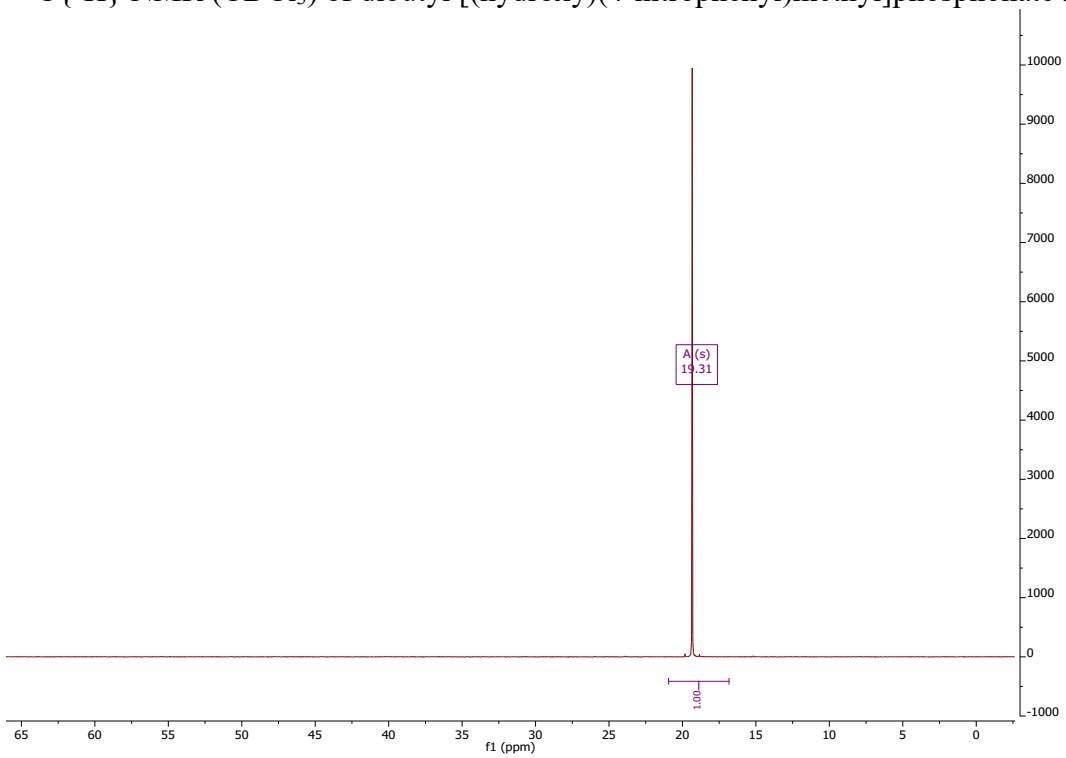
$^{31}P\{^{1}H\}$ NMR ($CDCl_3$) of dibutyl[(hydroxy)(4-nitrophenyl)methyl]phosphonate **3**



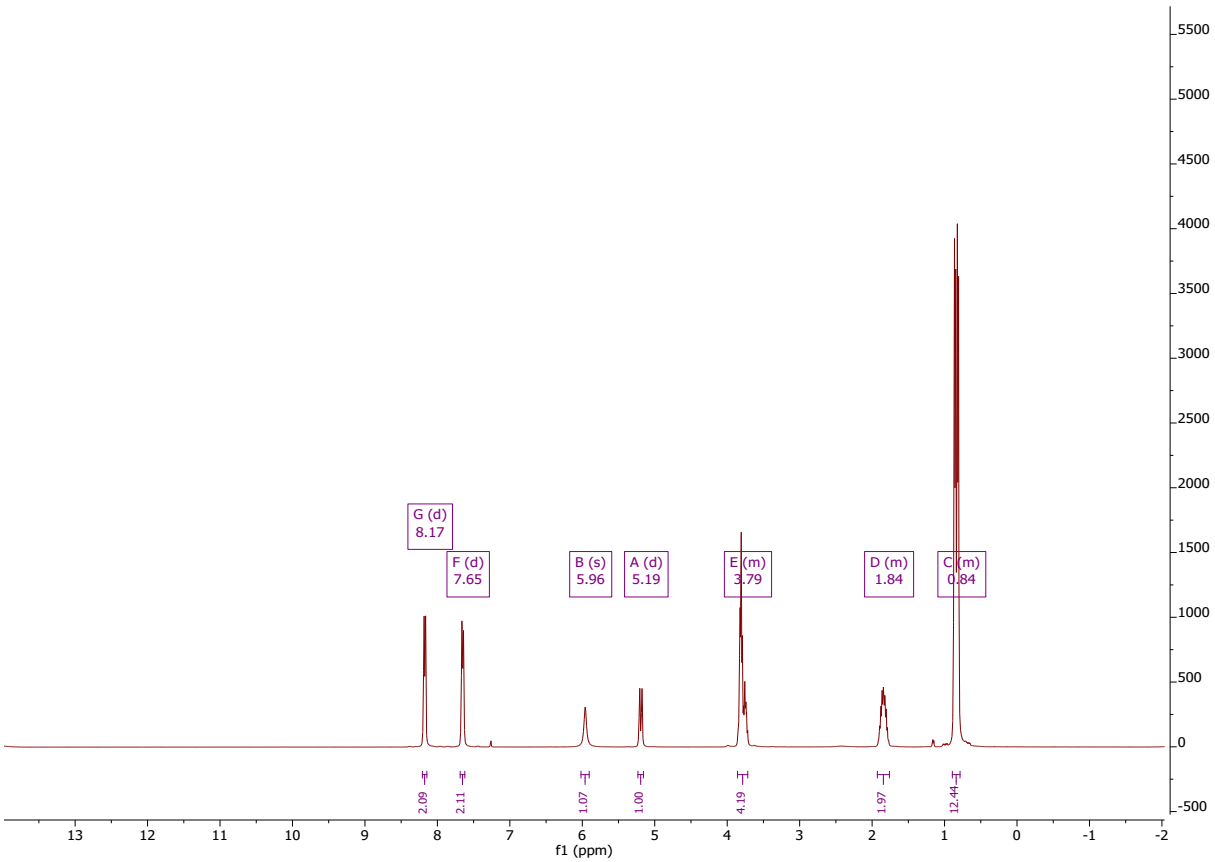
^{1}H NMR ($CDCl_3$) of [(hydroxy)(4-nitrophenyl)methyl]phosphonate **3**



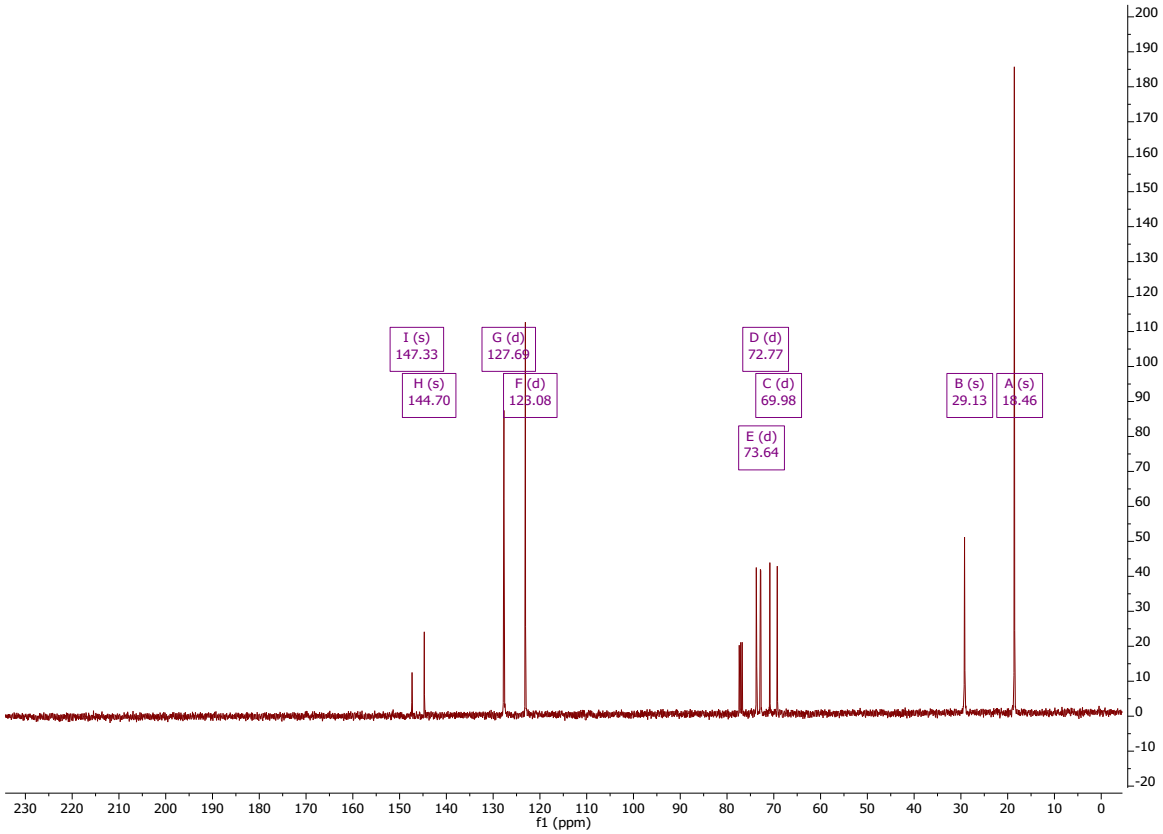
$^{13}C\{^{1}H\}$ NMR ($CDCl_3$) of dibutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **3**



$^{31}P\{^{1}H\}$ NMR ($CDCl_3$) of diisobutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **4**

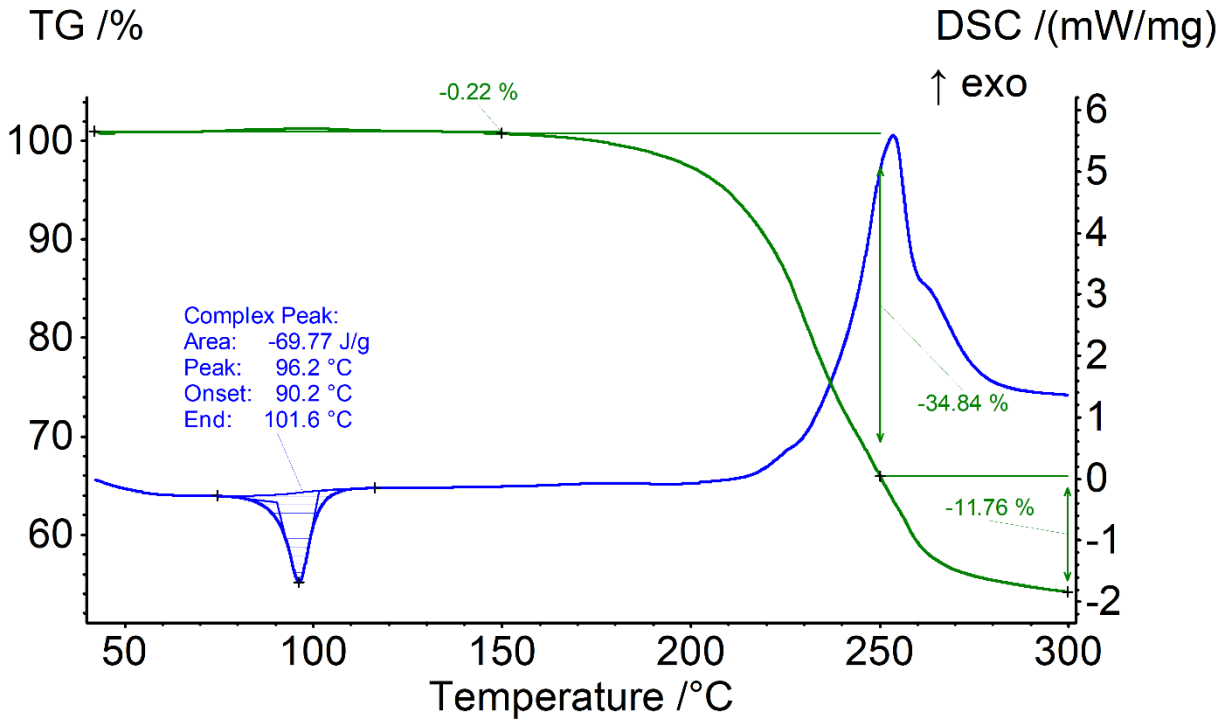


^{1}H NMR ($CDCl_3$) of diisobutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **4**

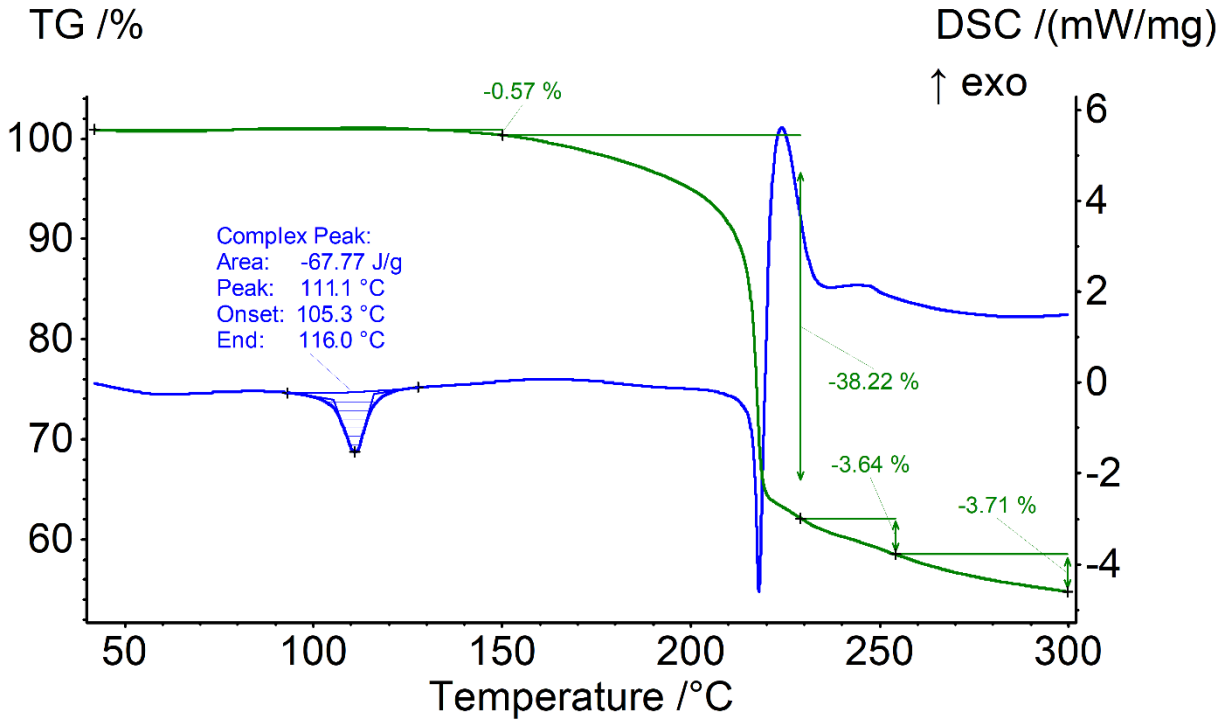


^{13}C{^{1}H} NMR ($CDCl_3$) of diisobutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **4**

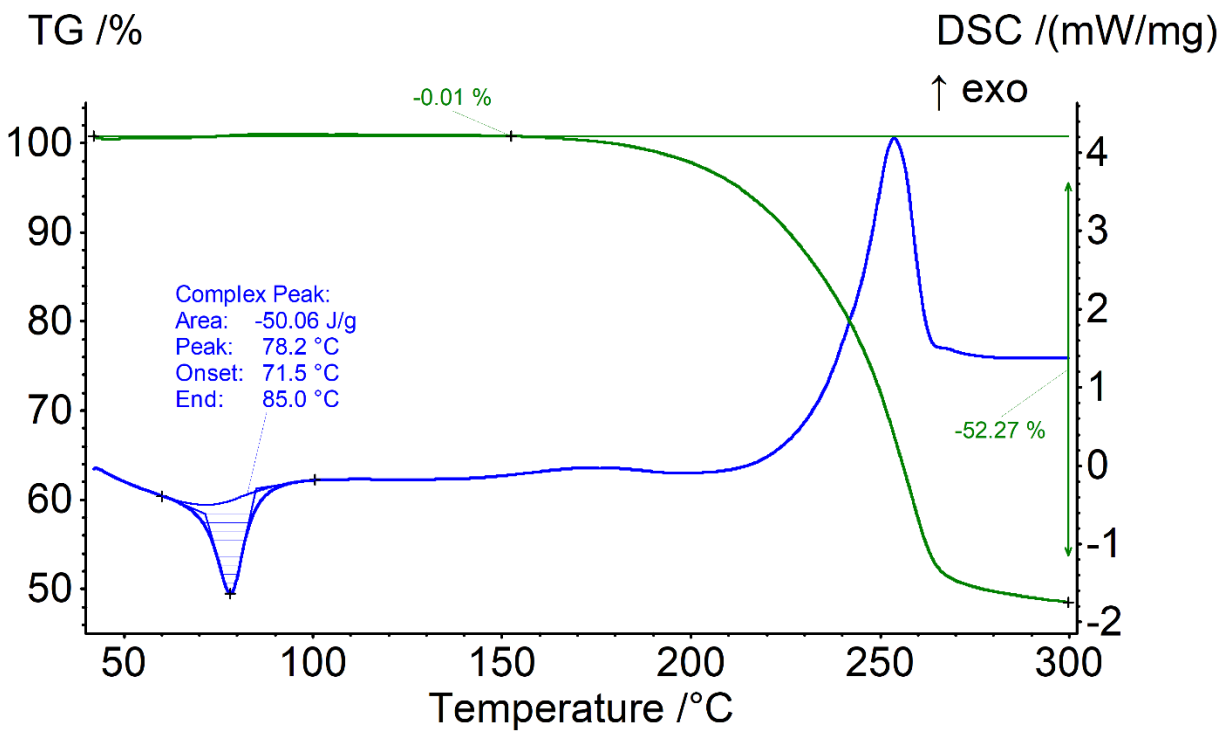
TG/DSC curves



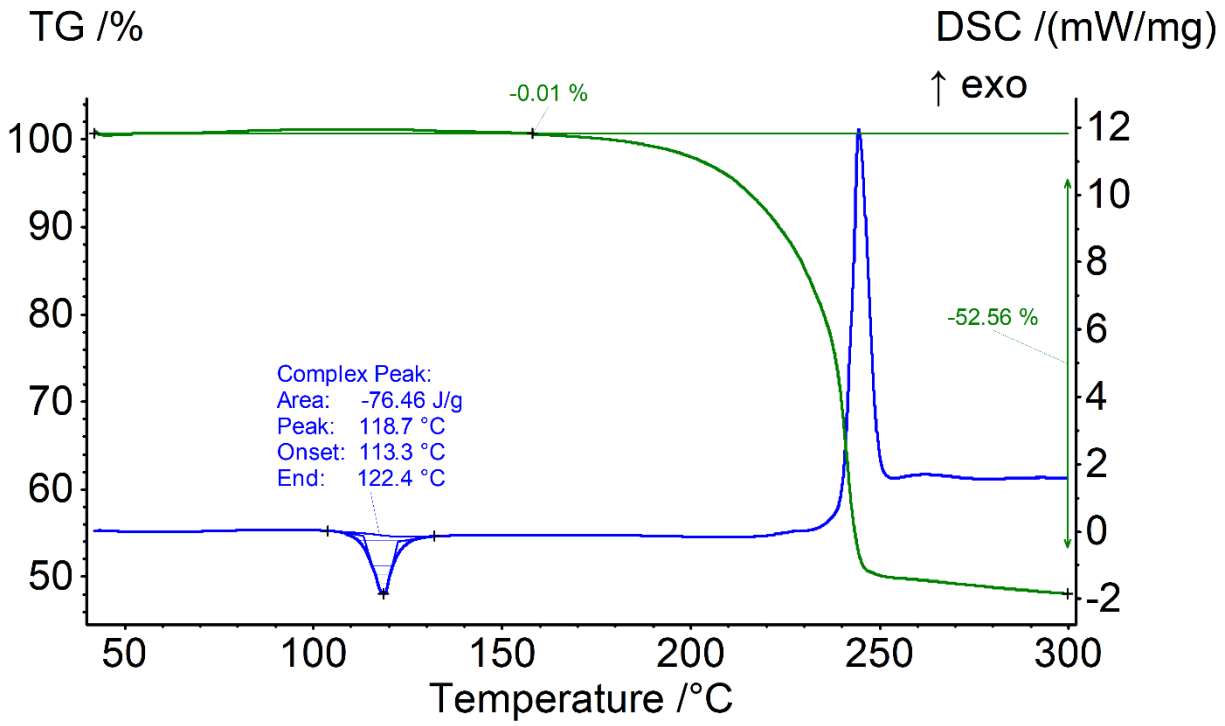
TG/DSC curves of diethyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **1**



TG/DSC curves of diisopropyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **2**



TG/DSC curves of dibutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **3**



TG/DSC curves of diisobutyl [(hydroxy)(4-nitrophenyl)methyl]phosphonate **4**

III. The details of hydrogen bonds and selected geometric parameters for dialkyl [(hydroxy)(4-nitrophenyl)methyl]phosphonates 1, 2 and 4

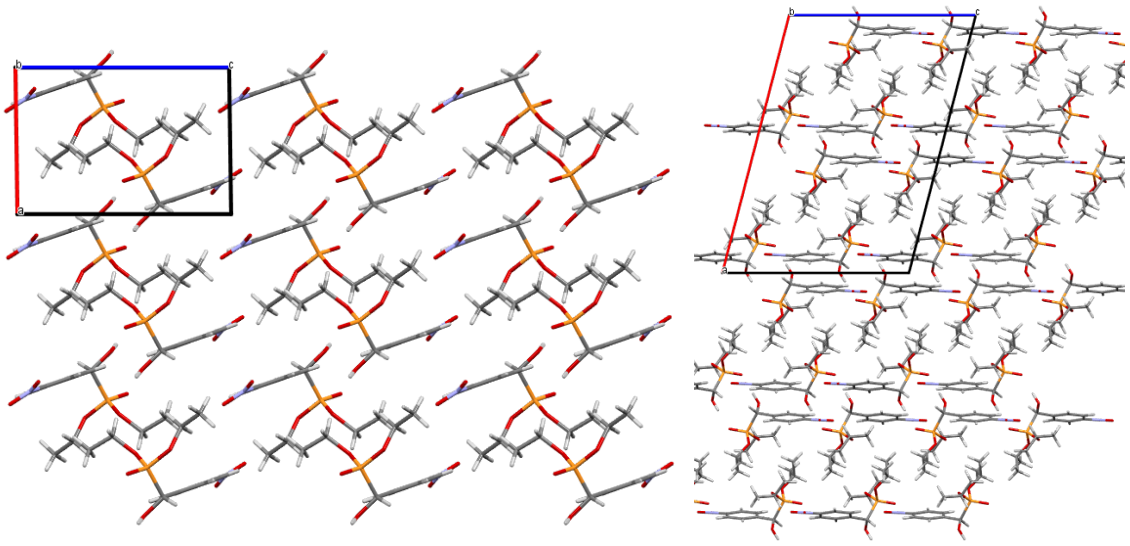


Figure S2. Crystal packing fragments in compounds **1** (left) and **2** (right).

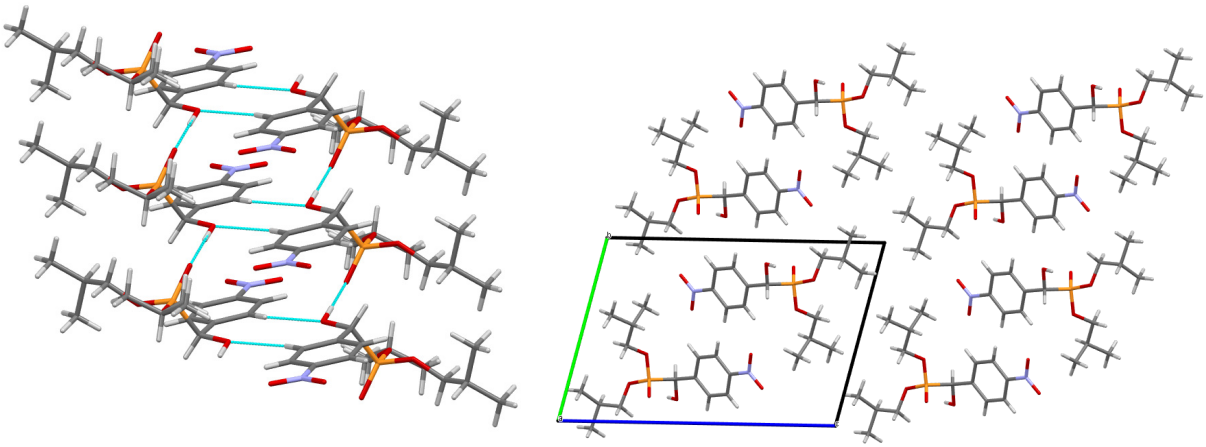


Figure S3. A fragment of crystal packing in compound **4**.

Table S1 Hydrogen bonds for compounds **1, 2** and **4**.

Compound	D-H···A	Symmetry code	D–H, Å	H···A, Å	D···A, Å	∠DHA, deg
1	O4–H4···O1	*–x*, 2*–y*, 1*–z*	0.93(3)	1.74(3)	2.664(2)	171(3)
2	O4–H4···O1	1*–x*, *y*, 1/2*–z*	0.90(3)	1.79(2)	2.680(2)	172(2)
4	O4–H4···O1	1+*x*, *y*, *z*	0.85(3)	1.85(3)	2.690(2)	173(3)

Table S2. Bond lengths [Å] and angles [°] for **1**.

P(1)-O(1)	1.4711(13)	C(2)-C(1)-C(6)	120.67(16)
P(1)-O(3)	1.5629(12)	C(2)-C(1)-H(1)	119.7
P(1)-O(2)	1.5669(14)	C(6)-C(1)-H(1)	119.7
P(1)-C(7)	1.8254(18)	C(3)-C(2)-C(1)	118.27(16)
O(2)-C(8)	1.449(2)	C(3)-C(2)-H(2)	120.9
O(3)-C(10)	1.468(2)	C(1)-C(2)-H(2)	120.9
O(4)-C(7)	1.409(2)	C(4)-C(3)-C(2)	122.37(16)
O(4)-H(4)	0.93(3)	C(4)-C(3)-N(1)	119.19(16)
O(5)-N(1)	1.226(2)	C(2)-C(3)-N(1)	118.44(16)
O(6)-N(1)	1.224(2)	C(3)-C(4)-C(5)	118.73(17)
N(1)-C(3)	1.466(2)	C(3)-C(4)-H(4A)	120.6
C(1)-C(2)	1.384(2)	C(5)-C(4)-H(4A)	120.6
C(1)-C(6)	1.392(2)	C(4)-C(5)-C(6)	120.32(16)
C(1)-H(1)	0.9500	C(4)-C(5)-H(5)	119.8
C(2)-C(3)	1.381(2)	C(6)-C(5)-H(5)	119.8
C(2)-H(2)	0.9500	C(5)-C(6)-C(1)	119.60(16)
C(3)-C(4)	1.378(3)	C(5)-C(6)-C(7)	120.48(15)
C(4)-C(5)	1.385(3)	C(1)-C(6)-C(7)	119.91(15)
C(4)-H(4A)	0.9500	O(4)-C(7)-C(6)	109.37(14)
C(5)-C(6)	1.388(2)	O(4)-C(7)-P(1)	108.80(12)
C(5)-H(5)	0.9500	C(6)-C(7)-P(1)	112.73(12)
C(6)-C(7)	1.513(2)	O(4)-C(7)-H(7)	108.6
C(7)-H(7)	1.0000	C(6)-C(7)-H(7)	108.6
C(8)-C(9)	1.488(3)	P(1)-C(7)-H(7)	108.6
C(8)-H(8A)	0.9900	O(2)-C(8)-C(9)	108.32(15)
C(8)-H(8B)	0.9900	O(2)-C(8)-H(8A)	110.0
C(9)-H(9A)	0.9800	C(9)-C(8)-H(8A)	110.0
C(9)-H(9B)	0.9800	O(2)-C(8)-H(8B)	110.0
C(9)-H(9C)	0.9800	C(9)-C(8)-H(8B)	110.0
C(10)-C(11)	1.493(3)	H(8A)-C(8)-H(8B)	108.4
C(10)-H(10A)	0.9900	C(8)-C(9)-H(9A)	109.5
C(10)-H(10B)	0.9900	C(8)-C(9)-H(9B)	109.5
C(11)-H(11A)	0.9800	H(9A)-C(9)-H(9B)	109.5
C(11)-H(11B)	0.9800	C(8)-C(9)-H(9C)	109.5
C(11)-H(11C)	0.9800	H(9A)-C(9)-H(9C)	109.5
		H(9B)-C(9)-H(9C)	109.5
O(1)-P(1)-O(3)	115.44(7)	O(3)-C(10)-C(11)	109.37(15)
O(1)-P(1)-O(2)	110.83(8)	O(3)-C(10)-H(10A)	109.8
O(3)-P(1)-O(2)	108.26(7)	C(11)-C(10)-H(10A)	109.8

O(1)-P(1)-C(7)	112.13(8)	O(3)-C(10)-H(10B)	109.8
O(3)-P(1)-C(7)	103.52(7)	C(11)-C(10)-H(10B)	109.8
O(2)-P(1)-C(7)	106.01(8)	H(10A)-C(10)-H(10B)	108.2
C(8)-O(2)-P(1)	125.60(11)	C(10)-C(11)-H(11A)	109.5
C(10)-O(3)-P(1)	122.41(10)	C(10)-C(11)-H(11B)	109.5
C(7)-O(4)-H(4)	104.5(17)	H(11A)-C(11)-H(11B)	109.5
O(6)-N(1)-O(5)	123.60(17)	C(10)-C(11)-H(11C)	109.5
O(6)-N(1)-C(3)	118.53(16)	H(11A)-C(11)-H(11C)	109.5
O(5)-N(1)-C(3)	117.87(16)	H(11B)-C(11)-H(11C)	109.5

Table S3. Bond lengths [Å] and angles [°] for **2**.

P(1)-O(1)	1.4733(12)	C(4)-C(3)-C(2)	122.44(15)
P(1)-O(3)	1.5612(11)	C(4)-C(3)-N(1)	119.11(15)
P(1)-O(2)	1.5711(12)	C(2)-C(3)-N(1)	118.43(15)
P(1)-C(7)	1.8198(15)	C(2)-C(1)-C(6)	120.41(15)
O(3)-C(11)	1.4664(19)	C(2)-C(1)-H(1)	119.8
O(2)-C(8)	1.468(2)	C(6)-C(1)-H(1)	119.8
O(4)-C(7)	1.4140(19)	O(4)-C(7)-C(6)	112.11(12)
O(4)-H(4)	0.90(3)	O(4)-C(7)-P(1)	104.67(10)
O(5)-N(1)	1.2303(19)	C(6)-C(7)-P(1)	114.09(10)
O(6)-N(1)	1.2266(19)	O(4)-C(7)-H(7)	108.6
N(1)-C(3)	1.464(2)	C(6)-C(7)-H(7)	108.6
C(6)-C(5)	1.389(2)	P(1)-C(7)-H(7)	108.6
C(6)-C(1)	1.389(2)	C(3)-C(4)-C(5)	118.28(15)
C(6)-C(7)	1.513(2)	C(3)-C(4)-H(4A)	120.9
C(3)-C(4)	1.378(2)	C(5)-C(4)-H(4A)	120.9
C(3)-C(2)	1.382(2)	C(4)-C(5)-C(6)	120.52(15)
C(1)-C(2)	1.379(2)	C(4)-C(5)-H(5)	119.7
C(1)-H(1)	0.9500	C(6)-C(5)-H(5)	119.7
C(7)-H(7)	1.0000	C(1)-C(2)-C(3)	118.59(15)
C(4)-C(5)	1.385(2)	C(1)-C(2)-H(2)	120.7
C(4)-H(4A)	0.9500	C(3)-C(2)-H(2)	120.7
C(5)-H(5)	0.9500	O(3)-C(11)-C(12)	109.63(14)
C(2)-H(2)	0.9500	O(3)-C(11)-C(13)	105.78(14)
C(11)-C(12)	1.500(3)	C(12)-C(11)-C(13)	113.18(15)
C(11)-C(13)	1.505(3)	O(3)-C(11)-H(11)	109.4
C(11)-H(11)	1.0000	C(12)-C(11)-H(11)	109.4
C(8)-C(10)	1.502(3)	C(13)-C(11)-H(11)	109.4
C(8)-C(9)	1.505(3)	O(2)-C(8)-C(10)	107.85(15)

C(8)-H(8)	1.0000	O(2)-C(8)-C(9)	106.76(15)
C(13)-H(13A)	0.9800	C(10)-C(8)-C(9)	113.45(17)
C(13)-H(13B)	0.9800	O(2)-C(8)-H(8)	109.6
C(13)-H(13C)	0.9800	C(10)-C(8)-H(8)	109.6
C(9)-H(9A)	0.9800	C(9)-C(8)-H(8)	109.6
C(9)-H(9B)	0.9800	C(11)-C(13)-H(13A)	109.5
C(9)-H(9C)	0.9800	C(11)-C(13)-H(13B)	109.5
C(12)-H(12A)	0.9800	H(13A)-C(13)-H(13B)	109.5
C(12)-H(12B)	0.9800	C(11)-C(13)-H(13C)	109.5
C(12)-H(12C)	0.9800	H(13A)-C(13)-H(13C)	109.5
C(10)-H(10A)	0.9800	H(13B)-C(13)-H(13C)	109.5
C(10)-H(10B)	0.9800	C(8)-C(9)-H(9A)	109.5
C(10)-H(10C)	0.9800	C(8)-C(9)-H(9B)	109.5
		H(9A)-C(9)-H(9B)	109.5
O(1)-P(1)-O(3)	116.01(7)	C(8)-C(9)-H(9C)	109.5
O(1)-P(1)-O(2)	114.53(7)	H(9A)-C(9)-H(9C)	109.5
O(3)-P(1)-O(2)	103.22(6)	H(9B)-C(9)-H(9C)	109.5
O(1)-P(1)-C(7)	112.33(7)	C(11)-C(12)-H(12A)	109.5
O(3)-P(1)-C(7)	103.70(7)	C(11)-C(12)-H(12B)	109.5
O(2)-P(1)-C(7)	105.86(7)	H(12A)-C(12)-H(12B)	109.5
C(11)-O(3)-P(1)	123.12(10)	C(11)-C(12)-H(12C)	109.5
C(8)-O(2)-P(1)	122.58(11)	H(12A)-C(12)-H(12C)	109.5
C(7)-O(4)-H(4)	107.1(16)	H(12B)-C(12)-H(12C)	109.5
O(6)-N(1)-O(5)	123.13(15)	C(8)-C(10)-H(10A)	109.5
O(6)-N(1)-C(3)	118.28(15)	C(8)-C(10)-H(10B)	109.5
O(5)-N(1)-C(3)	118.59(14)	H(10A)-C(10)-H(10B)	109.5
C(5)-C(6)-C(1)	119.71(14)	C(8)-C(10)-H(10C)	109.5
C(5)-C(6)-C(7)	120.23(14)	H(10A)-C(10)-H(10C)	109.5
C(1)-C(6)-C(7)	120.04(14)	H(10B)-C(10)-H(10C)	109.5

Table S4. Bond lengths [Å] and angles [°] for **4**.

P(1)-O(1)	1.4683(18)	C(2)-C(3)-C(4)	122.3(2)
P(1)-O(2)	1.5674(17)	C(2)-C(3)-N(008)	119.6(2)
P(1)-O(3)	1.5726(16)	C(4)-C(3)-N(008)	118.1(2)
P(1)-C(7)	1.822(2)	C(5)-C(4)-C(3)	118.3(2)
O(2)-C(8)	1.448(3)	C(5)-C(4)-H(4A)	120.8
O(3)-C(12)	1.465(3)	C(3)-C(4)-H(4A)	120.8
O(4)-C(7)	1.415(3)	C(4)-C(5)-C(6)	120.9(2)
O(4)-H(4)	0.85(3)	C(4)-C(5)-H(5)	119.5
O(5)-N(008)	1.223(3)	C(6)-C(5)-H(5)	119.5

O(6)-N(008)	1.220(3)	C(1)-C(6)-C(5)	119.4(2)
N(008)-C(3)	1.468(3)	C(1)-C(6)-C(7)	120.9(2)
C(1)-C(6)	1.387(3)	C(5)-C(6)-C(7)	119.6(2)
C(1)-C(2)	1.391(3)	O(4)-C(7)-C(6)	110.24(19)
C(1)-H(1)	0.9500	O(4)-C(7)-P(1)	107.55(16)
C(2)-C(3)	1.379(3)	C(6)-C(7)-P(1)	109.51(15)
C(2)-H(2)	0.9500	O(4)-C(7)-H(7)	109.8
C(3)-C(4)	1.384(3)	C(6)-C(7)-H(7)	109.8
C(4)-C(5)	1.379(3)	P(1)-C(7)-H(7)	109.8
C(4)-H(4A)	0.9500	O(2)-C(8)-C(9)	109.4(2)
C(5)-C(6)	1.394(3)	O(2)-C(8)-H(8A)	109.8
C(5)-H(5)	0.9500	C(9)-C(8)-H(8A)	109.8
C(6)-C(7)	1.510(3)	O(2)-C(8)-H(8B)	109.8
C(7)-H(7)	1.0000	C(9)-C(8)-H(8B)	109.8
C(8)-C(9)	1.496(4)	H(8A)-C(8)-H(8B)	108.2
C(8)-H(8A)	0.9900	C(8)-C(9)-C(10)	113.0(3)
C(8)-H(8B)	0.9900	C(8)-C(9)-C(11)	108.3(3)
C(9)-C(10)	1.523(5)	C(10)-C(9)-C(11)	110.8(3)
C(9)-C(11)	1.528(5)	C(8)-C(9)-H(9)	108.2
C(9)-H(9)	1.0000	C(10)-C(9)-H(9)	108.2
C(10)-H(10A)	0.9800	C(11)-C(9)-H(9)	108.2
C(10)-H(10B)	0.9800	C(9)-C(10)-H(10A)	109.5
C(10)-H(10C)	0.9800	C(9)-C(10)-H(10B)	109.5
C(11)-H(11A)	0.9800	H(10A)-C(10)-H(10B)	109.5
C(11)-H(11B)	0.9800	C(9)-C(10)-H(10C)	109.5
C(11)-H(11C)	0.9800	H(10A)-C(10)-H(10C)	109.5
C(12)-C(13)	1.500(3)	H(10B)-C(10)-H(10C)	109.5
C(12)-H(12A)	0.9900	C(9)-C(11)-H(11A)	109.5
C(12)-H(12B)	0.9900	C(9)-C(11)-H(11B)	109.5
C(13)-C(14)	1.506(4)	H(11A)-C(11)-H(11B)	109.5
C(13)-C(15)	1.530(3)	C(9)-C(11)-H(11C)	109.5
C(13)-H(13)	1.0000	H(11A)-C(11)-H(11C)	109.5
C(14)-H(14A)	0.9800	H(11B)-C(11)-H(11C)	109.5
C(14)-H(14B)	0.9800	O(3)-C(12)-C(13)	108.32(18)
C(14)-H(14C)	0.9800	O(3)-C(12)-H(12A)	110.0
C(15)-H(15A)	0.9800	C(13)-C(12)-H(12A)	110.0
C(15)-H(15B)	0.9800	O(3)-C(12)-H(12B)	110.0
C(15)-H(15C)	0.9800	C(13)-C(12)-H(12B)	110.0
		H(12A)-C(12)-H(12B)	108.4
O(1)-P(1)-O(2)	116.68(10)	C(12)-C(13)-C(14)	112.2(2)

O(1)-P(1)-O(3)	112.82(9)	C(12)-C(13)-C(15)	108.2(2)
O(2)-P(1)-O(3)	103.17(9)	C(14)-C(13)-C(15)	112.0(2)
O(1)-P(1)-C(7)	113.74(11)	C(12)-C(13)-H(13)	108.1
O(2)-P(1)-C(7)	101.38(10)	C(14)-C(13)-H(13)	108.1
O(3)-P(1)-C(7)	107.85(10)	C(15)-C(13)-H(13)	108.1
C(8)-O(2)-P(1)	122.07(16)	C(13)-C(14)-H(14A)	109.5
C(12)-O(3)-P(1)	120.79(14)	C(13)-C(14)-H(14B)	109.5
C(7)-O(4)-H(4)	106(2)	H(14A)-C(14)-H(14B)	109.5
O(6)-N(008)-O(5)	123.4(2)	C(13)-C(14)-H(14C)	109.5
O(6)-N(008)-C(3)	118.2(2)	H(14A)-C(14)-H(14C)	109.5
O(5)-N(008)-C(3)	118.3(2)	H(14B)-C(14)-H(14C)	109.5
C(6)-C(1)-C(2)	120.4(2)	C(13)-C(15)-H(15A)	109.5
C(6)-C(1)-H(1)	119.8	C(13)-C(15)-H(15B)	109.5
C(2)-C(1)-H(1)	119.8	H(15A)-C(15)-H(15B)	109.5
C(3)-C(2)-C(1)	118.6(2)	C(13)-C(15)-H(15C)	109.5
C(3)-C(2)-H(2)	120.7	H(15A)-C(15)-H(15C)	109.5
C(1)-C(2)-H(2)	120.7	H(15B)-C(15)-H(15C)	109.5